Li Gray

COMMONWEALTH OF AUSTRALIA.

DEPARTMENT OF NATIONAL DEVELOPMENT.

BUREAU OF MINERAL RESOURCES GEOLOGY AND GEOPHYSICS.

RECORDS. 1950/16

PETROLEUM TECHNOLOGY

Tentative METHODS OF TEST for porosity and permeability of rocks, including samples of drill cores.

TENTATIVE METHOD OF TEST FOR POROSITY OF ROCKS

including samples of Drill Cores.

Bureau of Mineral Resources, Geology and Geophysics.

Petroleum Technology Section

Method No. PT 2-49(T)

SCOPE

1. This method is intended for the determination of porosity of consolidated sediments. The method is applicable to those sediments included in rotary drill cores and hand specimens of rocks collected in the field. It is not applicable to sediments which are too incoherent to withstand normal careful handling, which might result in loss of sample in such cases, nor to samples showing irregular lithological variations.

Note 1. Results by this method tend to be slightly higher than those obtained by other methods which do not employ disaggregation of the sample to grain size or less.

DEFINITION:

2. (a) "Total porosity" is defined as the percentage of the total volume of the sample which is not occupied by the sample grains, cements, etc. This includes both connected and isolated pores, and is expressed in terms of volumes, as follows :

$$\text{Porosity} = 100 \ \frac{\text{pore volume}}{\text{bulk volume}} \ \%$$

where pore volume = bulk volume - grain volume.

The equation then becomes

$$\text{Porosity} = 100 \left(1 - \frac{\text{grain volume}}{\text{bulk volume}} \right) \%$$

OUTLINE OF METHOD:

3. The bulk volume of the extracted and dried sample is calculated from the weight of mercury displaced from a pycnometer. The grain volume is determined by crushing the sample to grain size or smaller and weighing in a pycnometer filled with kerosene of known density. The porosity is calculated by applying the equation in Section 2 (a).

APPARATUS:

4. (a) Chemical balance, accurate to 0.1 mgm.

(b) Suitably mounted hacksaw, capable of cutting the sample into a cube of edge approximately two centimetres.

(c) Grinding wheel

(d) Suitable wide-mouthed pycnometer, calibrated for different temperatures

(e) Porcelain pestle and mortar

(f) Soxhlet extraction apparatus

(g) Beaker, lightweight, squat form, 250 ml. capacity

(h) Balance case thermometer, accurate to 0.2°C.

MATERIAL :

5. (a) Kerosine, of known specific gravity at the temperature as determined in Section 8 (c).

SAMPLE:

6. (a) Preparation for Cutting:

A suitable fragment shall be warmed in a hot air oven to a temperature of 60 ± 5°C. It shall then be removed from the oven and, while hot, completely immersed in kerosene at room temperature and left for not less than one hour.

(b) Cutting and grinding :

Cut the kerosene-impregnated fragment into a cube of edge about 2 cm., using, as cutting fluid, kerosene of the same quality as used in impregnating the sample. Grind the edges of the cube so that they are replaced by approximately plane surfaces about 5 mm. wide. Use kerosene as a grinding fluid if necessary. No large angular cavities should be left on the surface of the sample.

(c) Wash off the cuttings by agitating the sample in kerosene, and extract the sample in the Soxhlet apparatus at least six times with acetone. Extract next with benzol at least six times in the same way, transfer to an oven, and dry at 95°C. for at least two hours. Transfer to a dessicator, and

allow to cool to room temperature.

PROCEDURE TO DETERMINE BULK VOLUME.

7. (a) Brush the sample with a camel-hair brush to remove loose fragments

(b) Fill the pycnometer in a beaker with mercury, brush the excess off the outside of the pycnometer and return the mercury to storage . Weigh the 250 ml. beaker to the nearest milligram (W_1), place the mercury-filled pycnometer in it, remove the cap and place this in the beaker also.

(c) Transfer the sample from the weighing bottle to the pycnometer, collecting the displaced mercury in the beaker, and brush the excess off the pycnometer. Replace the cap and, with the finger tightly over the cap, up-end the pycnometer with a rolling motion to allow any air entrapped on the surface of the sample to escape. Replace the pycnometer in the beaker and remove the finger from the cap. Brush any mercury off the finger tip into the beaker. Fill the weep-hole of the pycnometer cap by introducing mercury with a tube drawn out to a capillary. Transfer the beaker, with the pycnometer in it, to the balance case, and allow to stand for twenty minutes to come to the temperature of the air. The temperature, as indicated by the thermometer in the balance case after ten minutes, is taken as the temperature T_1, and the volume of the pycnometer and the density, D_m, of mercury shall be ascertained at this temperature.

Note 2: During the up-ending of the pycnometer, the minimum area of its surface should be touched by the fingers, to avoid heating of the mercury and its subsequent expansion.

(d) At the expiration of 20 minutes, brush any mercury from the surface of the pycnometer into the beaker, remove the pycnometer altogether from the beaker, and weigh. to the nearest milligram, the beaker containing the mercury displaced by the sample, (W_2).

(e) The difference of the weighings, $W_2 - W_1$, gives the weight of mercury, W_m, displaced by the sample.

PROCEDURE TO DETERMINE GRAIN VOLUME:

8. (8) Remove the sample from the pycnometer, and brush off all adherent mercury. Transfer to the mortar and carefully grind the sample completely to grain size or smaller, avoiding any loss of sample or chipping of the mortar. Weigh the pycnometer to the nearest milligram, W_3.

(b) Transfer the crushed material to the pycnometer and weigh to the nearest milligram, W_g.

(c) Slowly add kerosene (Section 5 (A)) to it, while stirring with a fine wire to remove all entrapped air bubbles. Completely fill the pycnometer with kerosene, stand in a beaker, replace the cap, fill the beaker with kerosene so that the pycnometer is completely covered and cover with a watch glass.

(d) Put the beaker in the balance case and leave for 20 minutes to attain the temperature thereof. This shall be taken as the temperature recorded by the balance case thermometer, immersed in the kerosene in the beaker, ten minutes after the beaker and pycnometer are put in. This shall be the temperature of test, T_2.

(e) After 20 minuted the pycnometer is removed from the beaker, quickly and thoroughly wiped dry and weighed to the nearest milligram, W_k.

CALCULATION AND REPORT:

9. (a) Calculate the porosity as follows :-

$$P = 100\left(1 - \frac{D_m}{W_m}\left(V_p - \frac{W_k - W_g}{D_k}\right)\right)\%$$

where D_m - density of mercury at temperature T_1^o,

D_k = density of kerosene at temperature T_2^o,

V_p = volume of pycnometer at temperature T_2^o,

W_g = weight of pycnometer + crushed material,

W_k = weight of pycnometer + crushed material + kerosine and

W_m = weight of mercury displaced by the sample

(b) Express results to the first decimal place.

PRECISION :

10. (a) Owing to the nature of the test, no duplicate tests are possible. However, samples from adjacent portions of the original should give porosities comparable to within 5% of the mean.

TENTATIVE METHOD OF TEST FOR PERMEABILITY

(of Rocks, including Samples of Drill Cores.)

P.T. 1-49 (T).

SCOPE.

1. This method is intended for the determination of permeability. It is applicable to suitably sized samples of rocks and any other substances whose constitutions permit of their being treated by the procedure set out in this method, subject to their own inherent limitations relative to this method. It is not applicable if there are obvious lithological variations in the actual sample under test.

DEFINITIONS.

2. (a) "Permeability" is the reciprocal of resistance to the viscous flow of unit quantity of a fluid of unit viscosity in unit time through a unit cube of material. Permeability has the dimensions of an area,

$$\frac{(L^3\ T^{-1})\ (ML^{-1}\ T^{-1})\ (L)}{(L^2)\ (MLT^{-2})\ (L^{-2})} = (L^2)$$

in c.g.s. units, therefore, the unit of permeability is :

$$\frac{\text{1 ml./sec x 1 poise x 1 cm.}}{\text{1 sq. cm. x 1 dyne/sq.cm}}$$

(b) The American Petroleum Institute has tentatively adopted a unit of permeability called the "darcy", (plural "darcys"). This unit satisfies the dimensional criterion above, but is defined in terms as follows :

$$\text{1 darcy} = \frac{\text{1 ml./sec x 1 centipoise x 1 cm.}}{\text{1 sq.cm. x 1 atmosphere/sq.cm.}}$$

The customary unit is the "millidarcy", = 0.001 darcy, abbreviation "md".

OUTLINE OF METHOD

3. The rock sample is orientated with respect to the bedding planes, and ground to size by the procedure given in Section 6. It is then extracted with acetone and benzol, and dried in an oven. After cooling in a dessicator, it is mounted in the permeameter bomb as described in Section 7 (e), and

all connections made gas-tight in the apparatus. Dried air is then admitted, its flow being so regulated by a needle valve that the differential pressure, as shown by the manometer, is constant. The times for the passage of air through the sample are measured for varying differential pressures.

APPARATUS.

4. (a) Compressed air supply of at least 40 psi pressure

(b) Efficient drying tube

(c) Permeameter bomb as described in Appendix A.1 (a)

(d) Differential manometer, as described in Appendix A.16

(e) Dreschel type bottle, 500 ml. capacity minimum.

(f) Receiving vessel graduated to indicate 60 ml. content accurately.

(g) 2 Thermometers, accurate to, and graduated to 0.1°C. (I.P. 20 W are suitable.)

(h) Timer. The stop watch or other timing device used shall be graduated in divisions of 0.2 sec. or less, and shall be accurate within 0.1 per cent when tested over a thirty minute period.

(i) Pressure tubing, T-piece and screw-clip.

(j) Stands

(k) Diamond or other core-drill as specified in Appendix A.1. (c)

(l) Hacksaw mounted to cut drill cores into cylinders about 1-1/8" long.

(m) Micrometer screw guage; preferably metric; ratchet type.

MATERIALS

5. (a) Mercury for use in the manometer shall be chemically clean, and free of any material which might increas its vapour pressure above normal.

(b) Oil for use in the manometer shall have as low a vapour pressure as possible, and shall be free of more volatile fractions which might increase its vapour pressure above normal. A suitable grade is Apiezon 'B'.

(c) Benzol for extraction, completely free of oil and preferably double-distilled.

(d) Acetone for extraction, dried over calcium chloride for 24 hours, free from benzol, oil, and other organic impurities and preferably double-distilled. Boiling point 56.6°C. maximum.

SAMPLE

6. (a) Orientation. The sample shall first be orientated with respect to the bedding planes. If this be impossible the most likely direction shall be taken as the bedding plane, and core pencils cut parallel and perpendicular to the bedding, whether this be obvious or not.

(b) Grinding. A suitable fragment shall be warmed in a hot air oven to a temperature of not less than 55°C. nor more than 65°C. It shall then be removed from the oven and, while hot, immersed in kerosine at room temperature and left completely immersed for not less than one hour. On the expiration of this period, the fragment shall be drilled in the required direction with a diamond or other drill, thereby forming a core pencil. The length of this core pencil shall be such as to permit of its being cut to form a perfect right cylinder of the following dimensions -

Length - not less than 1-1/16" (2.70 cm.) nor more than 1-1/8" (2.86 cm.)

Diamater - not less than 55/64" (2.185 cm.) nor more than 57/64" (2.265 cm.)

Measurements of this cylinder shall be made with a micrometer screw gauge, using approved practice. Kerosine of exactly the same quality as used for soaking the sample shall be used for cooling and removal of cuttings. Over-heating in cutting, and sizing if necessary, shall be avoided to prevent the dehydration, subsequent shrinkage, and even fusion of minerals.

(c) Drying After sizing, if necessary, the core pencil shall be washed with kerosine, as free as possible of cuttings. It shall then be mounted in a suitable holder, e.g. a perforated rubber stopper, and at least 250 ml. of acetone sucked through it. Follow this by at least 250 ml. of benzol, after which the pencil shall be dried in the oven at 95°C. for at least two hours. Then allow it to cool in a desiccator to room temperature.

(d) Handling. Care in handling the ground and extracted core pencil is essential to avoid alteration in its measured dimensions.

PREPARATION OF APPARATUS

7. (a) The manometer; One side of the manometer shall be filled with mercury, the other with low vapour pressure oil. The tap on the manometer shall be turned so that the pressure differential is established initally on the mercury manometer.

(b) Connect the apparatus as shown in Fig.1, if necessary omitting only the actual bomb for its final position.

(c) All glass-to-glass joints shall be rendered air-tight with suitable grease. If evidence of leaking be noticed at any stage, the test shall be repeated, and previous results discarded.

(d) The amount of rubber tubing in the actual pressure system shall be kept to the minimum consistent with flexibility, in order to avoid contamination of the system by gases from the rubber.

(e) Remove the core pencil from the desiccator, position centrally in the core-holder and mount by pouring molten Woods metal round it. The metal shall completely cover the sides of the core pencil, and shall leave the plane ends completely bare. The core-holder shall then be mounted in the bomb, and the top screwed down tightly, using sound rubber gaskets as illustrated. Then connect the bomb into the pressure system with the core pencil vertical.

OPERATION OF APPARATUS.

8. (a) Turn on the air supply and adjust the needle valve, V, so that the difference of pressure, as shown on the manometer, is of a convenient magnitude. The air shall be permitted to flow through the core pencil for at least five minutes, to eliminate as far as possible any irregularities due to moist air absorbed by the core during the mounting in the bomb. During this time it is convenient to observe the manometer for evidence of irregular flow, and to form an estimate of the differential pressure necessary to give an acceptable flow-rate.

(b) When the core pencil has been subjected to the dried air flow for the specified time, and the flow is steady, the T-piece, T, shall be closed. Adjust the air pressure so that the water issues from the Dreschel bottle at the rate of about two drops per second. When the differential pressure, as registered on the manometer, becomes constant, slip the receiving vessel under the drop-wise stream of water, and start the timer at the instant that the first drop of water passes the 60 ml. graduation of the receiving vessel. As the meniscus of the water in the vessel passes this mark, stop the timer and record the time in seconds.

(c) The reading of the manometer immediately after complete efflux of water into the receiving vessel, shall be noted, and must correspond exactly with that noted immediately before starting the test, otherwise the test must be repeated, and the result discarded.

(d) During passage of the gas under the conditions of test, there shall be no variation in the differential pressure, as recorded on the manometer. Should there by any such variation, the test must be repeated.

(e) By adjusting the needle valve, alter the differential pressure as registered by the manometer, and repeat the operations in (b) and (c), so that at least three sets of results are obtained.

(f) The temperature of test shall be the mean of the temperatures, indicated by the thermometer strapped to the side of the bomb, at the commencement of passing air through the core pencil, at the completion of test and five minutes thereafter. Where several tests are run consecutively on the same sample, the temperature at the completion of each shall be recorded for averaging. Should the temperature vary by more than 1°C. at any time during these periods, the test must be discarded.

NOTE: The test should be conducted in a draught-free room, as far distant as possible from sources of heat which might change the air temperature.

(g) The time of the actual test run shall not be less than 20 seconds using the oil manometer, nor less than 30 seconds with the mercury manometer.

(h) For core pencils of very low permeability , the oil manometer should be used. In this case, the temperature of the oil shall be ascertained by means of a thermometer strapped to the middle of the part of the limb occupied by the oil on the low pressure side.

CALCULATION AND REPORT

9. (a) The permeability shall be expressed as millidarcys, and calculated from the following formula when the mercury manometer is used :

$$k = \frac{V \times h \times v \times 76000}{t \times a \times p}$$

where V = volume of air passing through the core (60 ml.)

h = length of core in c.m.

v = viscosity of air in centipoises at the temperature as determined in Section 8 (f).

t = time in seconds

a = cross-sectional area of the core in sq.cm., and

p = differential pressure in cm. of mercury.

(b) The permeability in millidarcys is calculated from the following formula when the oil manometer is used :

$$k = \frac{V \times h \times v \times 76000 \times d_o}{t \times a \times P_o \times d_m}$$

where V, h, v, t and a have the values assigned in Section 9 (a), and

P_o = differential pressure in cm. of oil,

d_o = Specific gravity of the oil at the temperature determined in Section 8 (g), and

d_m = specific gravity of mercury at the temperature of the oil.

NOTE: (i) v, d_o and d_m are determined from the appropriate accompanying charts.

(ii) The permeability as claculated above, is identical with "permeability" as denoted in Section 1, para. 7. of API Code on Determination of Permeability, API Code No.27, 2nd. Edition. April 1942.

(c) The report shall include the following information-

(i) Permeability = ... md parallel to the bedding

(ii) Permeability = ... md perpendicular to the bedding

Where the bedding is indeterminate, as specified in Section 6 (a), "(apparent)" shall be written after "bedding" in (i) and (ii) immediately preceding.

PRECISION.

10. Results on the same core at different times should not differ from the mean by more than the following amounts :

Permeability	Repeatability	Reproducibility

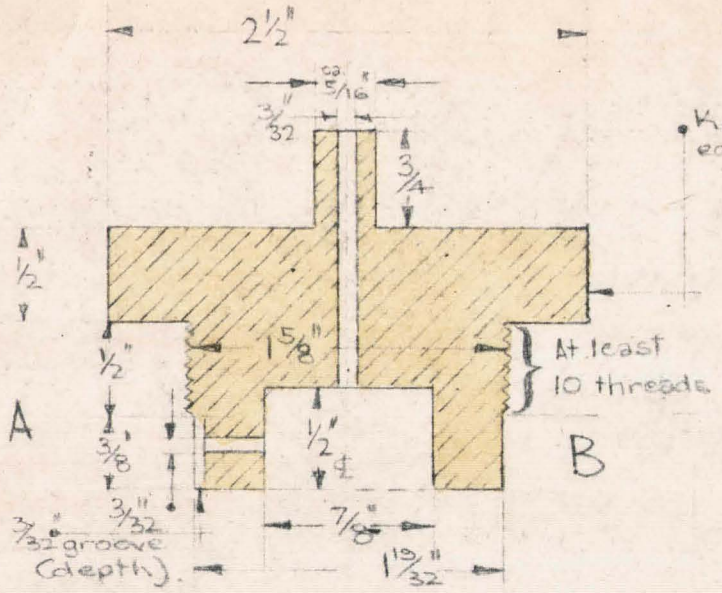
APPENDIX

A1. Apparatus

(a) Bomb - The bomb shall conform to the dimensions set out in Fig.2. The top of the bomb shall be composed of brass, and the thread shall comprise 26 turns to the inch. The core holder shall be turned from mild steel, and the bomb body from brass, of the same quality as the top of the bomb. The core holder shall be a slip fit in the bomb body. Tolerances shall be 0.1 millimeters, except where expressly stated as otherwise in the text of the Method. The assembled bomb shall appear as shown in Fig. 3.

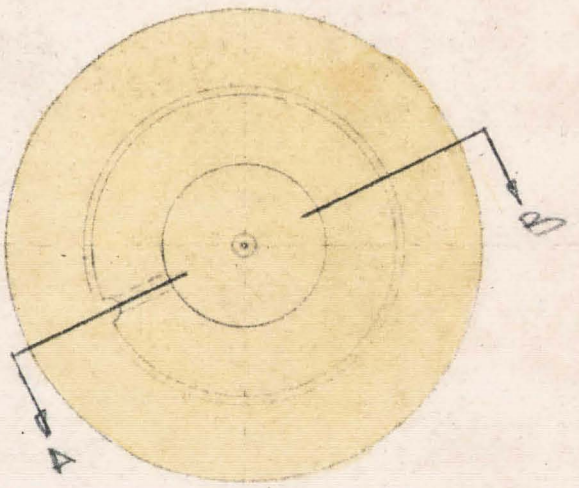
(b) The double differential manometer shall conform to the dimensions of the apparatus as set out in Fig. . Tolerances in all cases shall be 1 millimeter maximum.

(c) A diamond or other core drill, capable of cutting cylinders within the limits set out in Section 6 (b), and capable of being used safely with kerosine of flash point 90°F. minimum, as determined by ASTM D 93 - 46. Commercial power kerosine should be suitable as a coolant.

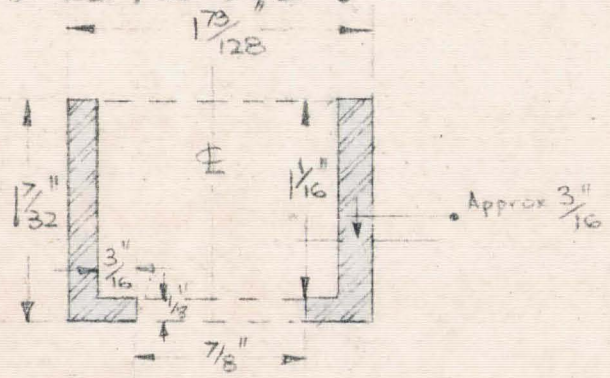


.TOP UNIT (BRASS).

.GROOVES TO BE 3/32" DEEP.

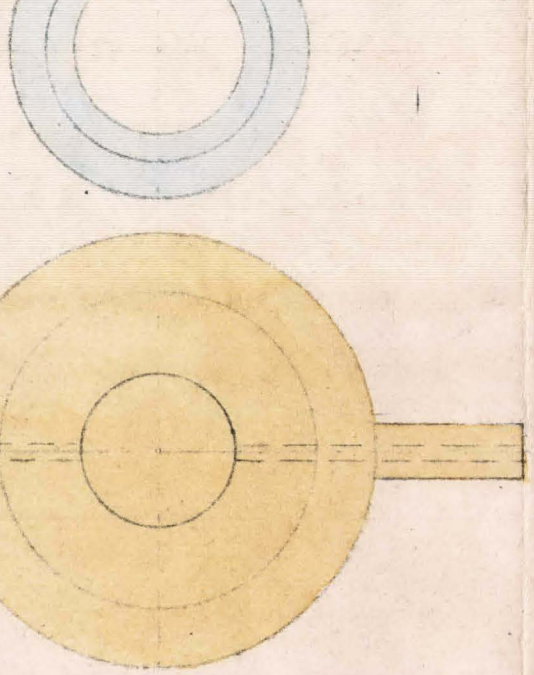


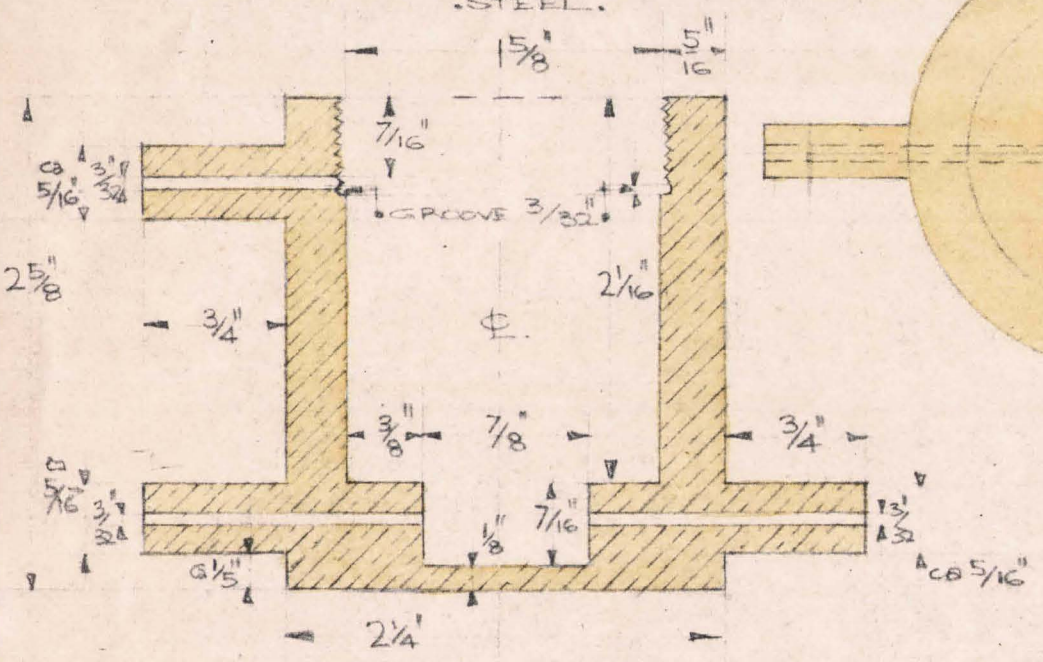
.LOWER VIEW.



.CORE HOLDER.

.STEEL.





.BOMB BODY.

.BRASS.

.SECTIONS. .PLANS.

NOTE TO TURNER: THE STEEL CORE HOLDER MUST FIT SNUGLY IN THE BRASS BOMB BODY. THE TOP MUST BE ABLE TO BE SCREWED DOWN TIGHTLY ON TO THE CORE HOLDER.

REFERENCE. N HOYLING 201049.

.BUREAU OF MINERAL RESOURCES GEOLOGY & GEOPHYSICS.
.PETROLEUM TECHNOLOGY SECTION.

.PERMEAMETER BOMB.

.NATURAL SCALE.

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